Metal Recycling from the Cathode of Lithium-Ion Battery in the Aqueous Environment

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Abstract. Lithium-ion battery recycling is crucial for conserving valuable materials, reducing environmental impacts, and ensuring a sustainable supply chain of critical metals for future energy storage technologies. However, establishing methods for selectively separating valuable metals through cost-effective, environmentally friendly processes is necessary to enhance the sustainability and efficiency of lithium-ion battery recycling. In this research, we developed an efficient and cost-effective method for selectively extracting precious, scarce metals—cobalt, manganese, and nickel—from NMC cathode-based batteries using a new combination of precipitation reactions. Firstly, some lithium-ion batteries with NMC cathodes were disassembled, and SEM and EDS were used to analyze a degraded battery to monitor its internal state. Then, two recycling approaches based on hydrometallurgical processes — acid leaching and step-by-step precipitation — were employed. In both methods, Co was removed as a complex with NO^{2-} ions. In other steps of the approaches, Mn and Ni were separated in different orders using NH_3 and NaOH as the simplest available precipitants. To characterize the separated batches, SEM, EDS, and XRD results confirmed the compositions. XRD results showed that Li was coprecipitated as a binary oxide with Mn ($LiMn_2O_4$, LMO) in the first approach. In the next approach, Li was detected as carbonate in a mixed salt coprecipitate. Additionally, the effect of precipitation resting time on the crystallinity of the batches was investigated. Also, the possibility of managing the unfavorable byproducts, such as NOx gases, in the Co separation step was examined.

Keywords: Lithium-ion battery, Recycling, NMC cathode, Hydrometallurgy, Precipitation

1 Introduction

Lithium-ion batteries (LIBs) have emerged as indispensable, widely adopted energy storage solutions in electric vehicles, especially in high-energy configurations. Nonetheless, their application extends beyond electric mobility and rapidly diversifies across other industries, including aerospace, the energy sector, and consumer electronics. The battery industry, particularly LIBs, relies on raw materials, including metals

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and various carbon allotropes, and has been eager to secure them. However, due to natural limitations, recycling and reusing spent products are inevitable [1-3].

As the global energy landscape shifts, the reliance on LIBs for these critical functions will continue to grow. However, this widespread adoption brings challenges, including resource constraints, environmental impacts from mining, and battery disposal issues. Addressing these challenges through sustainable production practices and efficient recycling methods will be crucial to ensuring that the benefits of LIBs can be realized without creating new environmental or resource-related issues [4-6].

Recycling of LIBs can have significant environmental impacts by reducing the consumption of natural resources, such as rock mines, and decreasing carbon footprints in supply chains. The total ecological impacts of industrial-scale production of battery-grade cathode materials from end-of-life LIBs, including greenhouse gas emissions, water consumption, and energy consumption, have been compared to those of conventional mining supply chains. Converting mixed-stream LIBs into battery-grade materials reduces environmental impacts by at least 58% [7]. Also, producing mixed-metal products rather than discrete salts can further reduce environmental impacts.

There are three main metallurgical approaches to LIB recycling. (1) Pyrometallurgy: This method employs high temperatures, typically exceeding 1000°C, to melt and reduce battery components into mixed alloys. As an established process, pyrometallurgy is similar to the techniques used for large-scale extraction of metals from ores. (2) Hydrometallurgy: This method leverages aqueous chemistry to selectively recover metals from LIBs, involving several steps to dissolve, purify, and isolate individual metals. (3) Biometallurgy: Still in its early stages, this approach utilizes microorganisms (bacteria and fungi) or their metabolic byproducts to extract metals from LIBs. It holds promise due to its environmentally friendly characteristics [8-10]. Other approaches to battery recycling include direct recycling and mechanical separation. Direct recycling focuses on recovering and refurbishing degraded or scrap electrodes without breaking them down to their elemental metal constituents [11]. Mechanical separation involves physical techniques such as crushing, sieving, and magnetic separation to isolate different battery components [12]. These methods offer alternative ways to recover valuable materials while minimizing the need for complex chemical processes. A comparison of the market values of worldwide LIBs recycling in 2022, with forecasts for 2026 and 2033, by process (in billion USD) is discussed [13]. The data shows a slight increase in the market size forecast from 2022 to 2026; however, the projections for 2033 seem overly optimistic. Some policymakers in developed countries have recently returned to previous policies to devalue and restrict electric vehicles. As a result, the recycling industry may oscillate and soon lose its value for a limited period, even though it is a rational and inevitable response to natural restrictions.

The focus of this research is on the hydrometallurgical approach. The overall approach begins with pretreatment, akin to pyrometallurgy, which involves discharging, dismantling, and separating battery components to prepare them for further processing [14]. The next step, leaching, employs acids (inorganic or organic) to dissolve metals from battery materials. The choice of acid and leaching conditions is crucial for optimizing metal dissolution. Following this, impurity removal is performed to eliminate undesirable metals, such as iron, aluminum, and copper, using precipitation or solvent extraction methods. The purified solution is then subjected to metal recovery, during which target metals can be selectively extracted via precipitation, solvent extraction, or electrodeposition. Finally, any residual lithium in the solution is typically recovered through precipitation, often as lithium carbonate or similar compounds, or by other advanced methods [15-17].

Chemical precipitation widely produces valuable metal compounds that are insoluble in leachate solutions. Typical precipitation chemicals include sodium hydroxide, oxalic acid, ammonium oxalate, phosphoric acid, and sodium carbonate. However, the library of precipitation agents is growing. The effectiveness of chemical precipitation is highly influenced by pH, concentration, and temperature, and these parameters must be carefully controlled to achieve optimal results. However, the transition metals nickel, cobalt, and manganese have similar chemical properties, and co-precipitation often occurs, making them difficult to separate. This method is favored for its simplicity, low cost, and minimal equipment requirements. Key considerations include selecting appropriate precipitants and determining optimal conditions to prevent precipitate dissolution, thereby ensuring efficient metal recovery [18-22]. The scope of this research is to introduce distinctive precipitation reactions, with the primary goal of recovering transition metals.

2 Experimental

2.1 Materials and Instruments

We mostly used Sigma-Aldrich, Acey, and Merck materials (lab or technical grades) for the overall acid leaching and metal recovery methods. Sulfuric acid (H_2SO_4, 98%), hydrogen peroxide (H_2O_2, 30%), spent cathode of LG (18650) Li-ion battery, filter papers in different sizes, sodium hydroxide (NaOH, 98%), sodium nitrite ($NaNO_2$), potassium nitrite (KNO_2), potassium chloride (KCl), dimethyl glyoxime (DMG), potassium hydroxide (KOH, 85%), potassium carbonate (K_2CO_3), ammonia solution (NH_3, 28-30%), ammonium fluoride (NH_4F), N-methyl-2-pyrrolidone (NMP), and distilled-deionized water were used.

A battery tester (Neware BTS8) was used for battery discharging. SPECORD 200 PLUS analytik jena UV-Vis Spectrophotometer, and field-emission scanning electron microscopy (FESEM, TESCAN MIRA III) equipped with an energy dispersive X-ray analyzer (EDS) were used to analyze electronic spectra, capture microscopic pictures, and semi-quantitative analysis of the materials produced in the process steps, respectively. X-ray diffraction (XRD) patterns were obtained using a powder diffractometer (Explorer, GNR) with Cu Kα radiation, a step size of 0.4°, and a 6-s integration time. The XRD technique was used to characterize the phase of the solid products.

2.2 Methods

The experimental section is planned based on the literature on the chemical reactions of metal cations [23]. The resource to initiate the processes was the cathode of a recharged and disassembled spent lithium-ion battery (LG brand). This was followed by stripping the cathode active material from aluminum foil using sonication with distilled water and NMP. The preferred solvent was water due to its safety and cost-effectiveness.

Leaching NMC in an acid solution results in the formation of several cations. Consequently, the presence of several cations in a solution complicates the separation process. Firstly, the focus was on total precipitation processes to separate the cations. Acid leaching was performed using a solution of H_2SO_4 (2 M) and H_2O_2 (10%). The ratio of acid solution volume to the mass of active cathode material was 30:1. The mixtures were stirred at 400 rpm and heated to 90˚C for up to 1 hour. During this time, the reactions were completed. Additional filtration was done to remove undissolved carbon content from the spent battery cathode source.

Procedure one. The overall process in this procedure (see Fig. 1) was based on leaching with the H_2SO_4/H_2O_2 pair and precipitating cobalt with KNO_2, manganese with an NH_3 solution, and nickel with DMG and NaOH [23]. The reagents were added in excess to expedite precipitation, while the solutions precipitated slowly. The primary estimate of the metal content in the mother liquor from the leached solution was determined by UV-Vis spectroscopy, following a reported method [24]. The absorption bands at 512 nm and 394 nm are associated with Co(II) and Ni(II), respectively. Estimation indicates that the leached solution has a Ni: Co ratio of 5:2, suggesting that the NMC cathode could have the (5:3:2) composition. It was confirmed later by EDS analysis of the cathode.

The resulting compounds were supposed to be $K_3[Co(NO_2)_6]$, MnO_2, $Ni(DMG)_2$, and $Ni(OH)_2$ in the process. However, deviations from our expectations, especially regarding Mn, were observed and discussed in the following sections. Also, potassium hexanitritocobaltate (III) reacted with heated NaOH to afford cobalt (III) hydroxide. The resulting samples were analyzed using SEM, EDS, and XRD.

Procedure two. The steps in this procedure (see Fig. 6) were similar to those in procedure one. However, the NH_3 solution was applied to precipitate nickel as nickel hydroxide, and in the last step, the K_2CO_3 reagent was used to check the possibility of precipitating lithium as lithium carbonate. In this process, manganese and nickel precipitates were immediately separated by filtration. Similar to procedure one, a sample of cobalt (III) hydroxide was rapidly prepared by alkaline hydrolysis and heating of the nitrite complex. The resulting samples were analyzed using SEM, EDS, and XRD.

3 Results and discussion

3.1 First Recycling Procedure

The first procedure for recycling metal cations is described in Fig. 1. This chart shows the order in which the first process was performed. Primarily, the acid leaching was done by H_2SO_4/H_2O_2 based on the reaction below [25]:

$Ni_xMn_yCo_zLiO_2$ (s) + 3/2 H_2SO_4 (aq) + 1/6 H_2O_2 (aq) → x $NiSO_4$ (aq) + y $MnSO_4$ (aq) + z $CoSO_4$ (aq) + 1/2 Li_2SO_4 (aq) + 5/3 H_2O (l) + 1/3 O_2 (g) (while: $x + y + z = 1$) (1)

In our case study, (x, y, z) were (0.5, 0.3, 0.2).

KNO_2 was added to the leachate for the first step, and the mixture was stirred. The reaction in this step is [33]:

$$\begin{aligned} &Co^{2+}\,(aq) + 7\,NO_2^-\,(aq) + 2\,H^+\,(aq) + 3\,K^+\,(aq) \rightarrow \\ &\rightarrow K_3[Co(NO_2)_6]\,(s) + NO\,(g) + H_2O\,(l) \end{aligned} \tag{2}$$

The resulting yellow solid was collected with filtration, and after drying, the $K_3[Co(NO_2)_6]$ phase was confirmed with XRD. The reaction selectively separates cobalt from the mixture in the presence of K^+. The major problem is the production of toxic NO_x gases as exhaust. However, we tried to collect them in the subsequent attempts of procedure two. Replacement of KNO_2 with a pair of $NaNO_2$ and KCl as sources of nitrite and potassium ions was tested, too. This replacement reduces the cost of cobalt separation but complicates the solution by introducing additional ions.

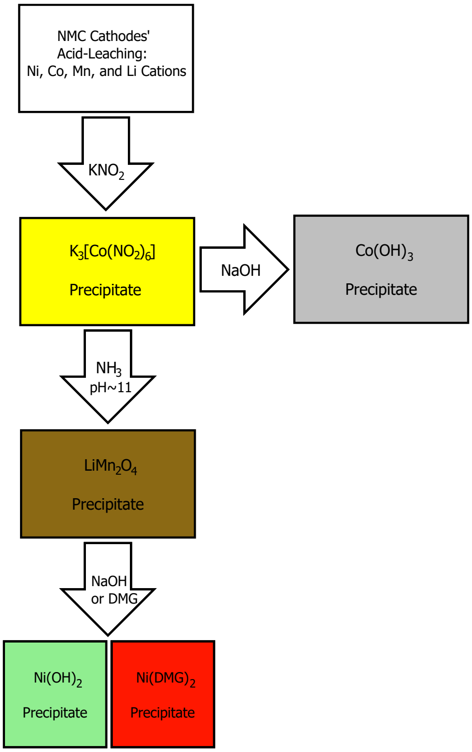


Fig. 1. The chart of the first procedure in the recycling of metal cations.

The precipitate was reacted with NaOH under heating and stirring to afford another valuable dark-brown cobalt compound, $Co(OH)_3$ [26]. The reaction is:

$$K_3[Co(NO_2)_6]\,(s) + 3\,NaOH\,(aq) \rightarrow Co(OH)_3\,(s) + 3\,KNO_2\,(aq) + 3\,NaNO_2\,(aq) \tag{3}$$

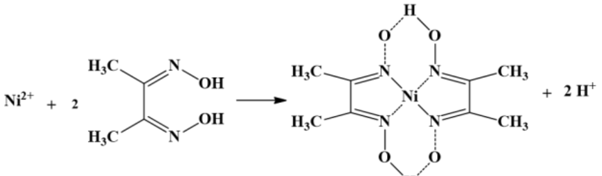
In the following steps, sufficient time was provided for each species to precipitate from the solution. In the second step, the ammonia solution was added to the filtrate, and at first, a light color of manganese precipitate was observable based on the reaction below:

$$Mn^{2+}\ (aq) + 2\ NH_3\ (aq) + 2\ H_2O\ (l) \rightarrow Mn(OH)_2\ (s) + 2\ NH_4^+\ (aq) \quad (4)$$

The addition of extra ammonia does not dissolve the manganese hydroxide. This reaction was gradually oxidized upon exposure to air, producing $MnO(OH)_2$ and MnO_2 species, which are insoluble in water and dark in color. During overnight rest, this step produced an unexpected phase ($LiMn_2O_4$) identified by XRD, which contained lithium. Manganese compounds precipitate while nickel remains in the solution since excess ammonia dissolves nickel hydroxide to produce an amine complex:

$$Ni^{2+}\ (aq) + 2\ NH_3\ (aq) + 2\ H_2O\ (l) \rightarrow Ni(OH)_2\ (s) + 2\ NH_4^+\ (aq) \quad (5)$$
$$Ni(OH)_2\ (s) + 6\ NH_3\ (aq) \rightarrow [Ni(NH_3)_6]^{2+}\ (aq) + 2\ OH^-\ (aq) \quad (6)$$

A DMG solution was prepared in ethanol/water (50:50) and added to a filtrate portion. A red precipitate of $Ni(DMG)_2$ was produced and filtered off. This color and precipitate have been recognized as a sign of complexation between Ni^{2+} and DMG ligands. So, the qualification analysis was canceled. The reaction is selective for nickel as:



(7)

Extra NaOH was added to the other portion of the nickel filtrate, which was then stirred overnight and left to rest for two days. The solution was then filtered and analyzed to characterize the precipitate.

In the final step, NH_4F was added to the filtrate to test for the availability of Li+ in the solution and to attempt its conversion to lithium fluoride precipitate (LiF). Afterward, only slight turbidity and no precipitate appeared in the solution. It seems that a substantial portion of the lithium was consumed earlier, and no results for the Li^+ cation batch alone were reported in the first procedure.

The analyses used to characterize the products at each step were SEM, EDS, and XRD. SEM images of the first product, $K_3[Co(NO_2)_6]$, are shown in Fig. 2a and reveal near-cubic crystalline structures. The XRD pattern for this step indicates the phase and shows the expected patterns for the potassium hexanitritocobaltate(III) complex. The semi-quantitative elemental analysis suggests it is free from Mn and Ni. So, it shows a successful cobalt separation. Also, SEM images of $Co(OH)_3$ are shown in Fig. 2b.

SEM images of the Mn batch after aging (Fig. 2c) reveal the intertwined sheet morphology. However, the XRD results show that the diffraction patterns are consistent with those of a $LiMn_2O_4$ species. Also, the semi-quantitative analysis shows the majority of Mn in the sample with negligible impurities of Ni and Co. The importance of the $LiMn_2O_4$ (LMO) species lies in its use as an active cathode material in some generations of lithium-ion batteries. Spinel LMO is a member of the intercalative class of compounds with 3-D lithium-ion transport possibility [27].

The SEM images of the Ni batch in Fig. 2d mention a well-shaped morphology. Similar to cobalt and manganese precipitation, nickel had enough rest to precipitate, resulting in a clear morphology. However, the semi-quantitative elemental analysis warns about manganese impurity.

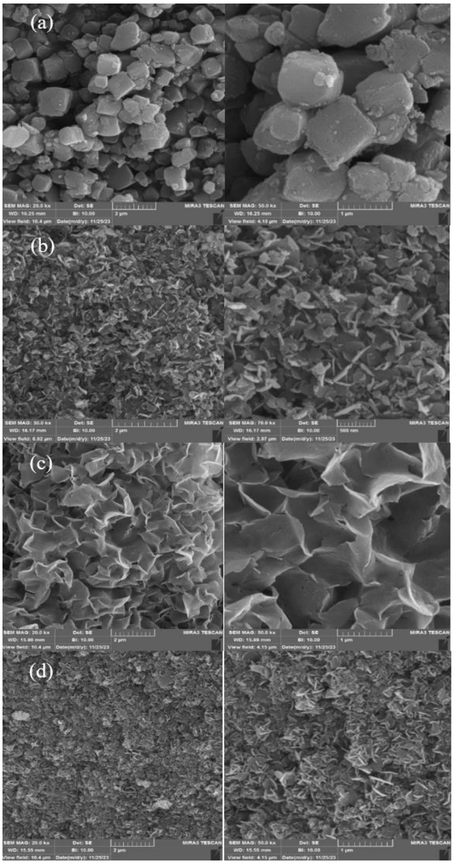


Fig. 2. The SEM images of cobalt complex, $K_3[Co(NO_2)_6]$ (a), cobalt (III) hydroxide, $Co(OH)_3$ (b), Mn batch (c), and Ni species (d) in different magnifications.

In conclusion, the process performed in the first procedure needs to rearrange the order of steps and check some other concepts, then try to use simpler precipitants that cancel the DMG for Ni precipitation, manage the gaseous wastes, use less solution volume, and employ fewer reagents to avoid the increase in the ionic strength of the final solution to ease the lithium extraction as a final batch.

3.2 Second Recycling Procedure

The second procedure for recycling metal cations is shown in Fig. 3. The first steps in this procedure, including acid leaching and cobalt separation, were the same as the last procedure, with a deviation to collect exhaust NO_x gases using a concentrated NaOH solution (and, in a minor case study, using an ammonia solution). Reactions between NO_x and NaOH take place in the aqueous phase. In the solution, NO_x first reacts with H_2O to form HNO_2 and HNO_3; thereafter, both HNO_2 and HNO_3 are neutralized with NaOH [28].

After cobalt separation, the nickel was precipitated with an ammonia solution, and the pH was then raised to form the soluble nickel hexaamine complex. For precipitation, a pH of 7-8 seemed appropriate. The solution was stirred and filtered to collect nickel hydroxide, as described in (5). The pH was then raised to ~11 with ammonia solution, yielding a precipitate of a manganese species. In this procedure, the tendency was to use fewer chemicals and lower solution volumes. It helps to increase the chance of lithium separation in the last step of the procedure. A saturated K_2CO_3 solution was added as a carbonate source to separate lithium as a lithium carbonate precipitate.

It is evident that among the vast diversity of compounds, only three of the lithium compounds could be categorized as insoluble [29], i.e., LiF, Li_2CO_3, and Li_3PO_4, and we used the proper reagents to precipitate Li as the first two solids. Lithium carbonate is slightly more soluble than the others (1.33 g/100 mL water), so for precipitation, we need more than the equivalent K_2CO_3 to force the reaction by a higher concentration of carbonate ions. It is noteworthy that the molar solubility of K_2CO_3 is more than that of Na_2CO_3, so that it can produce a higher concentration of carbonate ions in the solution.

In the first procedure, the solution settled overnight to complete the precipitation; however, separation was done in two hours in the second procedure. Results show that when the solution is left overnight, it has more time to become crystalline. However, for rapid separation, we observed an amorphous structure in the solids. The overnight settling of the Mn cation solution resulted in coprecipitation with Li cation, as observed in the XRD data. However, in this procedure, manganese and lithium precipitated in separate phases.

Analysis, including SEM, EDS, and XRD, was performed for each batch. The XRD pattern of the lithium batch is shown in Fig. 4. The batch pattern matches those of K_2SO_4 and Li_2CO_3 [30-31].

Since the acid leaching was performed using sulfuric acid, the counterion of the cations is the sulfate anion. Two sources of potassium, KNO_2 and K_2CO_3, were used in the process, and the precipitated Li_2CO_3 coprecipitated with K_2SO_4. The solubility of potassium salt

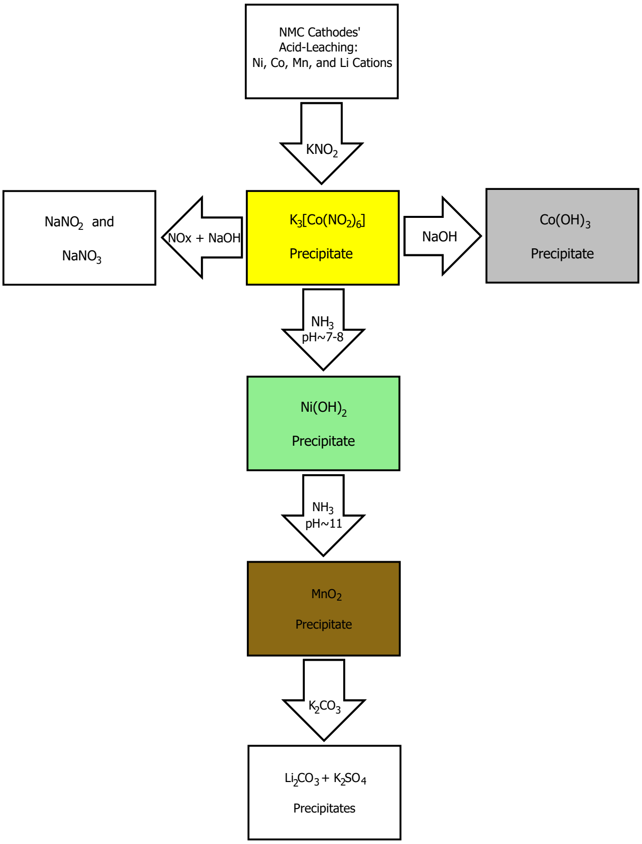


Fig. 3. The chart of the second procedure in the metal cations recycling.

as sulfate in 100 mL of water at 20°C is 11.1 g [29]. While its solubility is more than 8 times that of lithium carbonate under similar conditions, the K_2SO_4 concentration is high enough to coprecipitate.

SEM images of the lithium batch presented in Fig. 5a show two types of crystals: elongated prismatic K_2SO_4 crystals, reported in the literature to have formed by spontaneous nucleation [32], and round crystals, possibly Li_2CO_3. The EDS results indicate that the combination of two salts exists and that there are no impurities of Ni, Co, or Mn. There are reports about the purification of Li_2CO_3 from sulfate ions [33] using methods such as hydrogenation-decomposition procedures or ion-exchange steps with Dowex G26 resin (as an example). However, the developed procedures apply to situations where most of the batch consists of Li_2CO_3, with minor species being sulfate compounds. So, when the resulting solution is saturated with sulfate ions, an efficient lithium extraction process requires a revised procedure. Based on this result, the approach to recycling the lithium from the leached mother liquor needs improvement.

In the second procedure, cobalt was separated from the mixture as in the first procedure. Then, we followed and analyzed the steps that were done by deviation. To observe the low-aging-time effect, the complex solid was mixed with an excess amount of NaOH, boiled for 2 hours, and filtered immediately afterward. Fig. 5b shows the SEM image of the resulting solid of cobalt (III) hydroxide. No crystallinity was observed since the precipitate was separated without a resting time. XRD analysis confirmed the amorphous structure, as no peaks were observed. The EDS results for the batch showed no impurities of nickel or manganese.

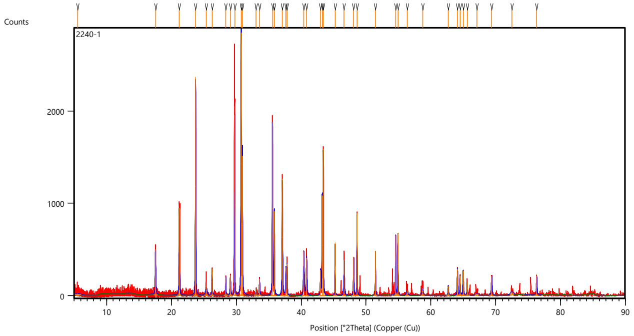


Fig. 4. The XRD pattern of the lithium batch.

The SEM image of the nickel batch in Fig. 5c shows no crystalline shapes or morphology, thanks to the immediate filtration of the resulting precipitate. Also, XRD analysis showed no peaks, confirming the absence of crystallinity. In the EDS results, the nickel batch lacks Mn and shows negligible Co impurities.

Finally, the last step in separating the transition metals from our battery resource was completed for Mn separation. The SEM image of the manganese batch (Fig. 5d) and EDS results confirm the predominant presence of Mn, with no XRD peak indicating an amorphous structure due to rapid filtration and a short aging time. However, an impurity of nickel was observed in the batch, which was attributed to the applied pH (~7-8), while no manganese was detected in the nickel batch. Since $Ni(OH)_2$ starts precipitating at the pH of 5.1 to become complete at 8.8, and for $Mn(OH)_2$, precipitation starts at 7.3 and completes at 10.1 [34], so there is a probable shared range of ~7.3-8 (in the applied pH) that both compounds can precipitate. It is a reason for the nickel impurity in the manganese batch. If raising the pH completes the nickel precipitation, manganese will be present as an impurity in the nickel batch due to the pH overlap discussed above. As a plan, it could be a reasonable experiment to test whether $Ni(OH)_2$ precipitation completion can be achieved at a pH below 7.3.

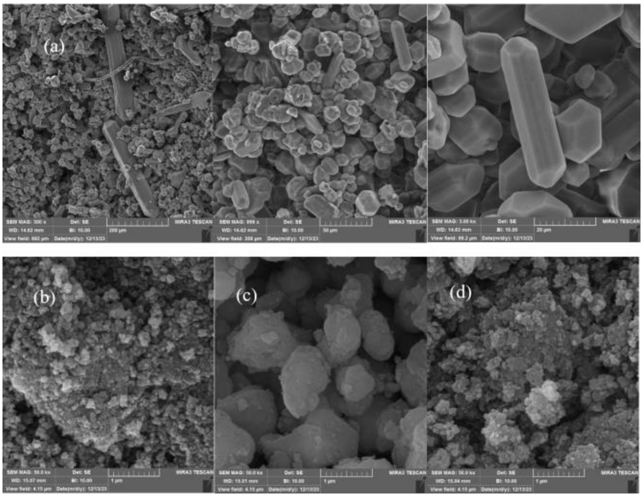


Fig. 5. The SEM images of (a) the lithium batch in different magnifications, (b) the cobalt (III) hydroxide batch prepared in a boiling alkaline situation with low rest time, (c) the nickel batch, and (d) the manganese batch.

4 Conclusion

In this research, we followed two precipitation processes as straightforward clues of the hydrometallurgical method. We tried to use inexpensive reagents, employ simple concepts, and implement achievable, straightforward processes. Our processes produced compounds such as $K_3[Co(NO_2)_6]$, $Co(OH)_3$, $Ni(OH)_2$, $Ni(DMG)_2$, MnO_2, $LiMn_2O_4$, and Li_2CO_3, which are valuable compounds of our available metals, Co, Ni, Mn, and Li. Moreover, no complex tools or processes are required. However, there are some intrinsic barriers to hydrometallurgy and, subsequently, to precipitation processes. Working with an increasing volume and, so, dilution of aqueous solutions that prevents the complete precipitation of favored species and necessitates the consumption of excess precipitants. These excess reagents, on the other hand, increase the solutions' ionic strength, thereby preventing additional precipitation. Fortunately, the release of NO_x gases appears manageable by using NaOH (or NH_3) solution in a circular recycling system. After all, development in metal recycling, especially in the energy storage sector, is required, and this kind of research can be a step forward in better addressing current problems in applied sciences and engineering.

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